

and the finished cyclodextrin particles are available. In contrast to the multi-stage processes, no intermediate products are produced in the single-stage process.

The particles can be produced by continuous fluidized bed spray granulation, for example according to EP-A 163 836, or by discontinuous fluidized bed spray granulation, for example according to EP-A 70 719. Further, a process such as disclosed in WO-A 97/16078 for production of flavor granules in a conventional discontinuous operated fluidized bed rotor granulator may be employed.

The invention is preferably carried out continuously. A continuous process is better suited for industrial production and exhibits shorter dwell times. For the same material throughput, bed content in the continuous process of the fluidized bed spray granulation is lower than in the case of a discontinuous process. Instead of allowing all particles to grow at the same time, in the continuous fluidized bed spray granulation only a small amount of particle nuclei are sprayed, and, following achievement of the desired particle size, they are immediately discharged, for example via an air separator. The encapsulated flavor and/or aroma cyclodextrin particles produced in accordance with the invention exhibit a small particle size distribution; besides this the particles (granules) can be removed selectively at the desired size.

In the framework of the present invention, it is preferred that the fluidized bed has a small bed height. Preferably this is 3 to 5 cm, in particularly preferred is 5 to 20 cm.

By continuous fluidized bed spray granulation, there results from the spray mixture free flowing, low dust, granular particles. Therein, in the ideal case, there occurs simultaneously in a granulation apparatus the basic processes of nucleus production, drying, formation and selective discharge of the particles that have reached the desired particle size.

The basic principle of continuous fluidized bed spray granulation (Chemical Engineering Technology, 62 year (1990), page 822 through 834) has been realized in countless variations. One can distinguish in particular the variants with external nuclei formation, in which nuclei from external sieves, grinders or other solids storage devices are dosed or metered into the bed, as well as variants with internal nuclei formation.

In the framework of the present invention, processes with internal nuclei supply are preferred. One of these is described for example in EP-A 163 836. This granulation device further utilizes a self-regulating mechanism for particle size regulation and thus involves minimal dwell time.

The spray mixture can be sprayed from below, from the side, or even from above into the fluidized bed. For separation of entrained solids from the exhaust, countless variants are possible, which can be distinguished by the separation process (for example cyclone or filter) or by the location of the separation step (within or outside of the granulation apparatus).

Finally, for the discharge of particles, air separators are preferably employed, for example as described in EP-A 332 031. With the air separators, it is accomplished that only the large particles are able to leave the fluidized bed. The remaining particles remain in the fluidized bed until they have reached the desired particle size.

For production of the inventive particles, first an aqueous spray mixture is produced. This spray mixture contains, besides water, at least one cyclodextrin, at least one flavor and/or fragrance substance as well as at least one cellulose ether.

From this spray mixture, the inventive particles with the particle size of greater than or equal to 50 μm are produced in one process step in a fluidized bed temperature.

Inventive particles have a particle size of 50 μm to 1000 μm . Preferred are particles with a particle size of 70 μm to 500 μm , and particularly preferred are 100 to 300 μm .

The characterization of the particle sizes is with respect to the particle diameter and is taken from the particle distribution curve. This particle distribution curve represents the dependency of the distribution sum $Q_3(x)$ of the particle diameters x . The distribution sum $Q_3(x)$ is the normalized total amount of all particles with a diameter smaller than or equal to x . The particle size $x_{50,3}$ is that particle size at which the distribution sum $Q_3(x)=0.5$. Unless otherwise indicated, all references to particle size in the present text are with respect to the particle size $x_{50,3}$. The index 3 indicates that the type of the measured amount is the volume (Ullmanns Encyclopedia of Technical Chemistry, Volume 3, Process Technology I, 4th Edition, Chemical Publications, Weinheim, 1972, pages 24-34).

The particle size determination via volume distribution can occur by laser diffraction (for example with the Master Sizer® MSS Longbench produced by Malvern Instruments Ltd., Malvern, UK).

Suitable cyclodextrins include alpha, beta, gamma as well as substituted cyclodextrins. Preferred are alpha, beta, gamma cyclodextrin or their mixtures, and preferred among these is beta-cyclodextrin. In a preferred embodiment, the cyclodextrin of the spray mixture is comprised solely of beta-cyclodextrin.

The proportion of cyclodextrin in the spray mixture is preferably 5 to 50 wt. %, preferably 10 to 40 wt. %, and particularly preferably 15 to 30 wt. %.

The proportion of water in the spray mixture is preferably 40 to 95 wt. %, more preferably at 50 to 90 wt. %, most preferably 60 to 80 wt. %.

The proportion of flavor and/or fragrance in the spray mixture is preferably 0.0005 to 15 wt. %, more preferably 0.5 to 10 wt. %, most preferably 0.25 to 5 wt. %.

The spray mixture contains, as granulation aid, a binder, namely cellulose ether. The cellulose ether is preferably selected from the group including methylcellulose, ethylcellulose, propylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, hydroxy-propylmethylcellulose, carboxymethylcellulose, carboxymethylhydroxyethylcellulose and ethylhydroxyethylcellulose; particularly preferred is carboxymethylcellulose (CMC).

The cellulose ether content of the spray mixture is preferably 0.1 through 6 wt. %, particularly preferably from 0.2 to 2 wt. %. The dried particles preferably contain between 0.5 and 10 wt. %, particularly preferably between 2 and 4.5 wt. % cellulose ethers.

Preferred are cellulose ethers with a viscosity of 15 to 200,000 mPas, more preferred are those with a viscosity of 1,000 to 50,000 mPas, most preferred are those with a viscosity of 5,000 to 15,000 mPas. The viscosity characteristics are determined with a 2 wt. % solution in water at 20° C.

As measuring system there is used a conical plate system with a diameter of 40 mm and a cone angle of 4°. The temperature is 20° C., the shear rate is 1 s^{-1} . The viscosity can be determined using, for example a rheometer CVO 120 (produced by Bohlin Instruments GmbH, Pforzheim, Germany).